भारतीय मानक
Indian Standard

IS 4566 : 2020

मिथाइलीन क्लोराईड (डिक्लोरोमेथेन) — तकनीकी की विशिष्टि

(दूसरा पुनरीक्षण)

Specification For Methylene Chloride (Dichloromethane) — Technical

(Second Revision)

ICS 71.080.20

© BIS 2020

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

June 2020 **Price Group 7**

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, After the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1968 and revised in 1979. In the first revision the requirements for acidity were modified for the industrial grade of the material. Also another grade suitable for the manufacture of refrigerants was added and the methods of tests was modified suitably.

This second revision, requirement of Purity has been specified and distillation range has been made optional for grade 1.Alternate method for heavy metal has been added and test method for impurity has been modified. Amendment Nos. 1 and 2 have also been incorporated.

Methylene chloride (dichloromethane) finds extensive use as a major component of non-flammable paint removers; in the manufacture of photographic films; solvent for alkaloids, bitumens, crude rubber, oils, resins, waxes and many organic compounds; as solvent mixtures for cellulose esters and ethers, textile and leather coatings, lacquers, fire-extinguishing compositions;

refrigeration; local anesthetic in dentistry; as spotting agent; degreasing and dewaxing agent; in chemical syntheses; as a vapour pressure depressant in aerosols; and various other uses in the chemical industry.

Though least toxic of the chloromethane, methylene chloride is strongly narcotic causing dizziness and slight nausea. It is very dangerous to the eyes. In view of this and its great volatility, care should be taken in its use. The place where methylene chloride is handled and stored should be well ventilated and it should not be brought in contact with hot surfaces and naked flame (*see also* **5.1.1, 5.2.1** and **5.2.2**).

The Indian standards listed in Annex C which are referred in this standard are informative in nature. All standards are subject to revision and parties to agreement based on standard are encouraged to apply the most recent editions of the standards.

The composition of the Committee, responsible for the formulation of this standard is given at Annex D.

For the purpose of whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SPECIFICATION FOR METHYLENE CHLORIDE (DICHLOROMETHANE) — TECHNICAL

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for methylene chloride (dichloromethane), technical.

2 REFERENCES

The following Indian standards contain provisions which through reference in this text constitute the provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1260 (Part 1) : 1973	Pictorial Marking For Handling And Labelling Of Goods : Part 1 Dangerous Goods (*First Revision*)
2362 : 1993	Determination of Water by Karl Fischer Method — Test Method *(Second Revision*)
2303 (Part 2) : 2018 / ISO 4802-1 : 2016	Grading Glass for Alkalinity Part 2 Hydrolytic Resistance of Glass Containers — Determination by Titration Method and Classification (*Second Revision*)

3 GRADES

3.1 The material shall have two grades, namely:

a) *Grade* 1 - for the use in photo film industry, and

b) *Grade* 2 - suitable for industrial and other applications.

4 REQUIREMENTS

4.1 Description

The material shall be a colourless liquid free from matter in suspension and shall have a sweet penetrating ether-like odour. It shall be soluble in ethyl alcohol and ethyl ether in all proportions.

4.2 The material shall also comply with the requirements prescribed in Table 1, when tested according to the methods given in Annex A. Reference to the relevant clauses of Annex A is given in col 5 of the Table No 1.

Table 1 Requirements for Methylene Chloride (Dichloromethane), Pure and Technical

(*Clause* 4.2)

Sl No.	**Characteristic**	**Requirement**		**Method of Test, Ref to Clause No. in Annex A**
		Grade 1 (Pure)	Grade 2 (Technical)	
(1)	(2)	(3)	(4)	(5)
i)	Relative density[1] at *27/27°C*	1.314 to 1.319[2]	1.310 to 1.320	A-2
ii)	Distillation range at a pressure of 1 bar (Optional for grade 1)	2 to 97 percent by volume shall distil between 39 to 40.5°C	2 to 97 percent by volume shall distil between 39 to 41°C	A-3
iii)	Residue on evaporation, percent by mass, *Max*	0.001	0.01	A-4
iv)	Moisture, percent by mass, *Max*	0.02	0.05	A-5
v)	Acidity (as HCl), percent by mass, *Max*	0.0003	0.001	A-6
vi)	Free chlorine	To pass test	To pass test	A-7

Table 1 (*Concluded*)

Sl No.	Characteristic	Requirement		Method of Test, Ref to Clause No. in Annex A
		Grade 1 (Pure)	Grade 2 (Technical)	
(1)	(2)	(3)	(4)	(5)
vii)	Aldehydes	To pass test	--	A-8
viii)	Iron (as Fe), parts per million, *Max*	0.04	--	A-9
ix)	Heavy metals (as Pb), parts per million, *Max*	1.0	--	A-10
x)	Resistance to potassium permanganate	To pass test	--	A-11
xi)	Nesslers reagent	To pass test	--	A-12
xii)	Resistance to sulphuric acid	To pass test	--	A-13
xiii)	Total impurities due to methyl chloride and chloroform, percent by mass, *Max*	0.1	--	A-14
xiv)	Purity, percentage, *Min*	99.5	-	A-14

5 PACKING AND MARKING

5.1 Packing

5.1.1 Subject to the provisions of the Red Tariff No. 18 of 1960 issued by the Indian Railways Conference Association with any additions and alterations made thereafter, the material shall be packed in fully air-tight well-closed galvanized iron containers capable of withstanding a pressure of 0.5 bar depending upon the maximum temperature to which it would be exposed. The containers shall have preferably replaceable closure. Any other suitable container compatible with the properties of the material may also be used.

NOTES

1 Relative density is the term adopted for specific gravity with water as reference substance by the International Organization for Standardization (ISO). This term is synonymous with specific gravity.

2 The correction factor within the range 15 to 35° is + 0.0016 for each degree Celsius fall and − 0.001 6 for each degree Celsius rise in temperature.

5.2 Marking

5.2.1 The containers shall be suitably marked in red letters as POISONOUS and shall be labelled as shown in Fig. 5 of IS 1260 (Part 1): 1973. The label shall also bear the following clear and legible instruction:

'STORE IN A COOL, WELL VENTILATED PLACE AWAY FROM FIRE AND FLAMES. USE WITH ADEQUATE VENTILATION. AVOID PROLONGED BREATHING OF VAPOURS'

5.2.2 The containers shall be marked with the name of the material; its net mass; name of the manufacturer and recognized trade-mark, if any;

5.2.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.'

6 SAMPLING

Representative samples of the material shall be drawn and their conformity to this standard shall be judged as prescribed in Annex B.

ANNEX A

(*Clause* 4.2 and *Table* 1)

METHODS OF TEST FOR METHYLENE CHLORIDE, PURE AND TECHNICAL

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the results of analysis

A-2 DETERMINATION OF RELATIVE DENSITY

A-2.0 General

Relative density is determined using specific gravity bottle or pyknometer.

A-2.1.1 *Specific Gravity Bottle or Pyknometer*

With well-fitting ground-glass joints. To calibrate, clean and dry the bottle or pyknometer thoroughly, weigh and then fill with water recently boiled and cooled to below 27°C after removing the cap of the side arm. Fill to overflowing by holding the bottle or pyknometer on its side in such a manner as to prevent entrapment of air bubbles, insert the stopper and immerse in a water bath at 27 ± 0·2°C. Keep the entire bulb completely covered with water and hold at that temperature for 30 min. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, replace the cap, cool to a lower temperature and weigh. Calculate the mass of water. The constant gravity bottle of about 50 ml capacity and of either of the two shapes as shown in Fig. 1 is recommended.

A-2.1.2 *Water Bath-maintained at 27 ± 0·2°C.*

A-2.1.3 *Thermometer*

Any convenient thermometer of a suitable range with 0.1 or 0.2°C subdivisions. The thermometer should be checked against a standard thermometer, which has been calibrated and certified by the National Physical Laboratory, New Delhi, or any other organization recognized for such work *(see* IS 4825).

A-2.2 Procedure

Fill the bottle or pyknometer with the material previously kept at about 27°*C* to overflowing, holding the apparatus on its side in such a manner as to prevent entrapment of air bubbles after removing the cap of the side arm. Insert the stopper, immerse in the water bath at 27 ± 0.2°C, and hold for 30 min. Carefully wipe off any liquid which has come through the capillary opening. Remove the apparatus from the bath, clean and dry it thoroughly. Replace the cap, cool to a lower temperature and weigh.

A-2.3 Calculation

$$\text{Relative density at } 27/27°\text{C} = \frac{\text{A-B}}{\text{C-B}}$$

where

A = Mass of the specific gravity bottle or pyknometer filled with the material at 27°*C,*

B = Mass of the specific gravity bottle or pyknometer, and

C = Mass of the specific gravity bottle or pyknometer filled with water at 27°C.

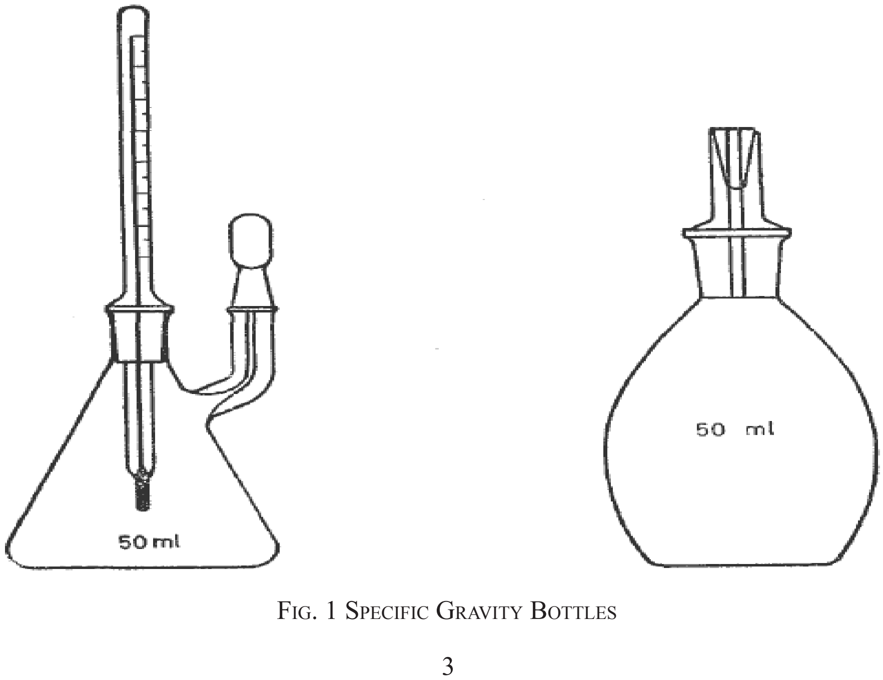


Fig. 1 Specific Gravity Bottles

A-3 DETERMINATION OF DLSTLLLATION RANGE

A-3.0 General

The fraction distilling at specified temperature range is collected and measured.

A-3.1 Apparatus

A-3.1.1 *Distillation Flask* — of the shape and dimensions shown in Fig. 2. Fix the flask in the vertical position by means of a clamp at the extreme upper end of the neck.

A-3.1.2 *Thermometer* — A suitable partial immersion thermometer having a range of 39 to 51ºC, graduated at each 0.2°C, with a maximum scale error of 0.2°C (*see* IS 4825).

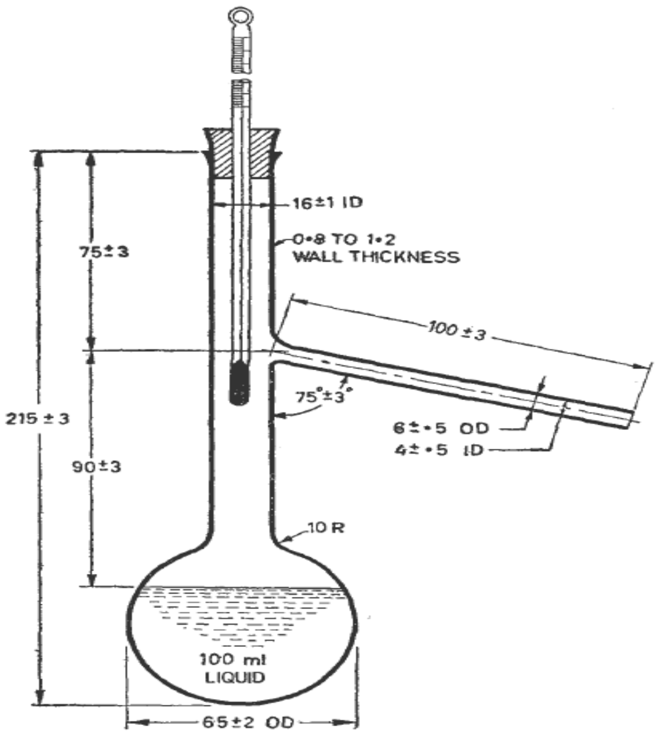
A-3.1.2.1 *Position of thermometer* — The thermometer shall be held concentrically in the neck of the flask by means of a well-fitting stopper of a material which is not attacked by the liquid, and the junction of the capillary tube and the main bulb of the thermometer shall be maintained level with the lower edge of the joint of the side-tube and the neck of the flask. The stopper shall project about 10 mm above the top of the neck of the flask. When the thermometer is fixed in position as indicated above, the immersion line on the thermometer shall be in the neighborhood of the top of the cork holding the thermometer.

A-3.1.2.2 The thermometer shall bear a certificate of the National Physical Laboratory, New Delhi, or any other organization recognized for such work.

A-3.1.3 *Rectangular Draught Screen,* made of 0.8 mm thick sheet metal, with the dimensions shown in Fig. 3 and open at the top and bottom, fitted with hard asbestos board, with a central hole of 100 mm diameter. It shall comply with the following requirements:

a) In each of the two narrow sides of the draught screen there shall be two circular holes, each 25 mm in diameter, and in each of the four sides of the draught screen there shall be three holes with their centres 25 mm above the base of the draught screen. These holes shall occupy the position shown in Fig. 3. The diameter of each of the holes centrally situated in the longer sides shall of 25 mm and of the remaining ten holes shall be 125 mm. At the middle of each of the wider sides a vertical slot with the dimensions shown in Fig. 3 shall be cut downwards from the top of the screen,

b) A sheet of hard asbestos, 6 mm in thickness, and having a central circular hole 50 mm in diameter, shall be supported horizontally in the screen and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos sheet may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.



All dimensions in millimetres

FIG.2 DISTILLATION FLASK

c) In one of the narrow sides of the screen a door shall be provided having the dimensions and position as shown in Fig. 3. In each of the narrow sides of the screen a mica window shall be placed centrally, with the bottom of the window on a level with the top of the asbestos shelf. The dimensions and position of the windows are shown in Fig. 3.

A-3.1.4 *Liebig Condenser* — with the bent end made of glass conforming to IS 2303 (Part 2) with a wall thickness of 1.0 to 1.5 mm and conforming to the shape and dimensions shown in Fig. 4.

A-3.1.4.1 Alternatively, the bent portion may be substituted by an adapter fitted externally in such a manner that distillate does not come in contact with the cork.

A-3.1.5 *Receiver or Graduated Cylinder* – 100 ml capacity, with millilitre marks running a halfway round the circumference, and 5 ml marks running three-quarters-way round and l0 ml marks running all round the circumference, and numbered as shown in Fig. 5. Any other graduated l00 ml cylinder may also be used.

A-3.1.6 *Water Bath, thermostatically controlled.*

A-3.1.7 *Half-Second Pendulum,* for measuring the rate of distillation.

A-3.2 Procedure

Assemble the apparatus as shown in Fig. 6. Measure 100 ml of the material at laboratory temperature by means of 100-ml receiver or graduated cylinder and transfer it to the distillation flask. Add a fragment (about 2 mm cube) of porous or other suitable inert material to prevent bumping. Connect the flask to the condenser and insert the thermometer. Pass an adequate supply of cooling water at 0 to 5°C through the condenser. To receive the distillate, use the receiver in which the sample was measured, without rinsing or drying. Heat the flask slowly especially after ebullition has begun, in order that the mercury column of the thermometer may become fully expanded before the first drop of distillate falls into the receiver, care being taken that the total period of this preliminary heating shall be for not less than 5 nor greater than 10 min. Place the receiver so that the condensate will flow down its side. Continue the distillation at the rate

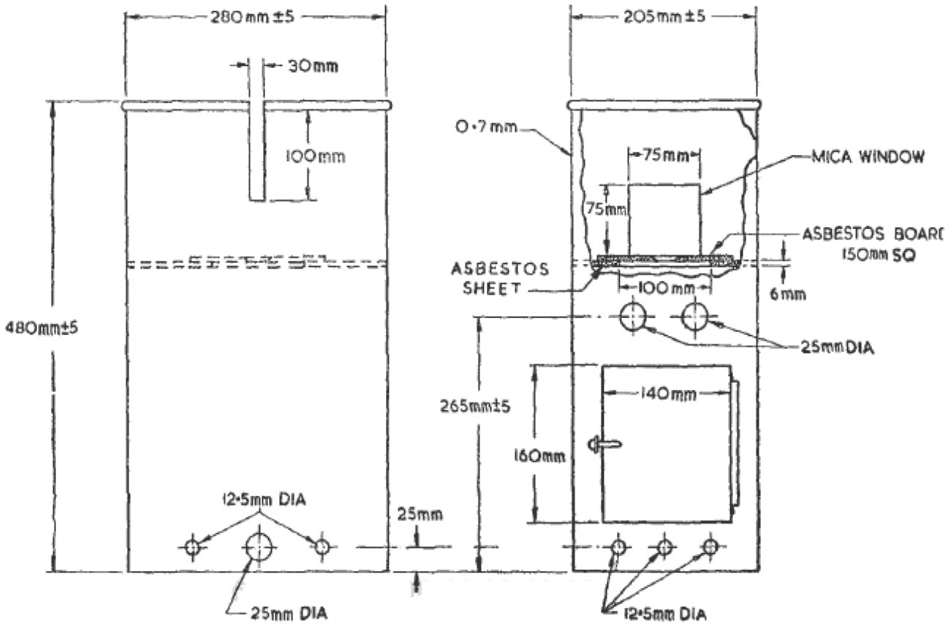


Fig. 3 Rectangular Draught Screen

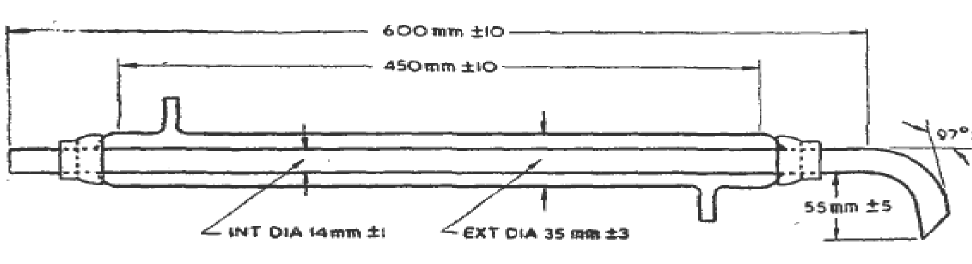


Fig. 4 Liebig Condenser

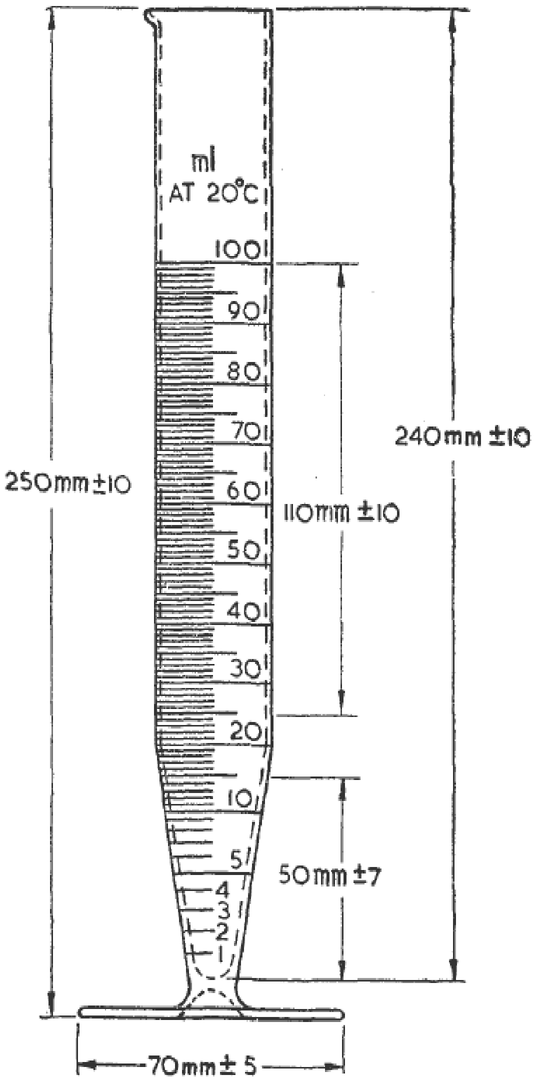


FIG. 5 RECEIVER OR GRADUATED CYLINDER

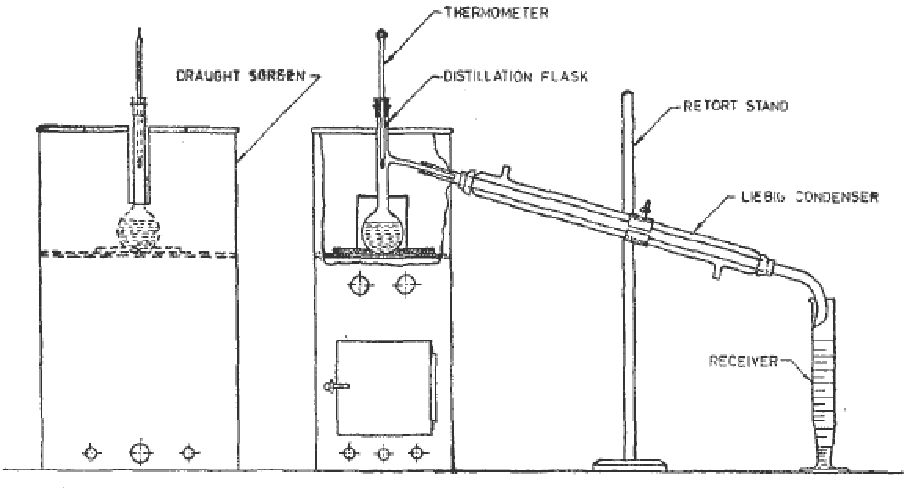


FIG. 6 ASSEMBLY OF APPARATUS FOR DETERMINING DISTILLATION RANGE

of 4 to 5 ml per minute (about 2 drops per second). Read the volume of distillate in the receiver when the thermometer indicates each of the specified distillation temperatures, the temperatures on the thermometer scale being corrected as specified under **A-3.3**. The difference between the volumes so recorded is the percentage by volume distilling between the specified temperatures at 760 mm pressure.

A-3.3 Correction of the Thermometer Reading

The following correction shall be applied before starting distillation.

A-3.3.1 *Error of Scale*

In all the thermometer readings, make the corrections as indicated on the certificate of the instrument.

A-3.3.2 *Correction for Barometric Pressure*

If the barometric pressure prevailing during the determination is normal, namely, 760 mmHg, no correction need be applied to the specified temperature and the thermometer scale as corrected under **A-3.3.1** shall be used as such. If, however, the prevailing pressure deviates from 760 mmHg, the specified temperature shall also be corrected as follows:

a) For every 10 mm above 760 mmHg pressure, subtract 0.38ºC from the observed temperature of the boiling range to get the specified temperature as specified in Table1.

b) For every 10 mm below 760 mmHg pressure, add 0.38ºC from the observed temperature of the boiling range to get the specified temperature range as specified in Table 1.

A-4 DETERMINATION OF RESIDUE ON EVAPORATION

A-4.1 Apparatus

A-4.1.1 *Porcelain Basin* — 150 ml capacity [*see* IS 2837 (Part 2)]

A-4.1.2 *Water-Bath*

A-4.1.3 *Oven,* capable of maintaining temperature between 105°C to 110°C.

A-4.2 Procedure

Clean the basin and heat in the oven at 105°Cto 110°C for 30 min and dry in desiccator. Weigh it to the nearest 0.000 2 g (M_1) after 30 min. Measure out 100 ml of the material in the basin (V). Evaporate the material over a water-bath in a fume cupboard to practically dryness. Further heat in the oven at 105°C to 110°C for 30 min. Cool in a desiccator for 30 min and weigh accurately. Repeat the heating for 15 min followed by cooling and weighing till constant weight is attained.

A- 4.3 Calculation

Residue on evaporation, percent by mass = $\frac{M}{d}$

Where

M = mass of the residue obtained, in g; and

d = relative density of the material.

A-5 DETERMINATION OF MOISTURE CONTENT

A-5.0 General

Moisture content is determined using the Karl Fischer method.

A-5.1 Take 50 ml of the material and determine the moisture content as described in IS 2362.

A-6 DETERMINATION OF ACIDITY

A-6.0 General

Acidity is determined by titrating a known quantity of material with a standard alkali.

A-6.1 Reagents

A-6.1.1 *Bromothymol Blue Indicator* — Dissolve 0.5 g of water soluble bromothymol blue crystal in l00 ml of water. Dilute 80 ml of this solution to 500 ml with methyl alcohol.

A-6.1.2 *Standard Sodium Hydroxide Solution*- 0.01 N.

A-6.2 Procedure

To 100 ml of water, neutral bromothymol blue, add 100ml of the material and shake vigorously for 1 min at least. Add bromothymol blue indicator and titrate with standard sodium hydroxide solution until a bluish green end- point persists for 1 min.

A-6.3 Calculation

Acidity (as HCl), percent

by mass = $3\cdot65\ \frac{V_1\ N}{V\ D}$

where

V_1 = volume in ml of standard sodium hydroxide solution,

N = normality of standard sodium hydroxide solution;

V = volume in ml of the material taken for the test; and

D = relative density of the material.

A-7 TEST FOR FREE CHLORINE

A-7.0 Outline of the Method

The material is shaken with a 3,3′-dimethylnaphthidine solution and the colour developed, if any, is noted.

A-7.1 Apparatus

A-7.1.1 *Graduated Measuring Cylinder*

50 ml, glass stoppered *(see* IS 878).

A-7.2 Reagent

A-7.2.1 3,3-*Dimethylnaphthidine Solution* — Dissolve 0.01 g of finely ground 3,3-dimethylnaphthidine in 5 ml of glacial acetic acid and dilute rapidly with water to 200 ml, Store the solution in the dark.

A-7.3 Procedure

To 50 ml of the material contained *in* the graduated measuring cylinder, add 5 ml of the 3,3-dimethylnaphthidine solution and shake the cylinder for 30 s.

A-7.3.1 The material shall be considered as showing no free chlorine; if no pink colour is developed.

A-8 DETERMINATION OF ALDEHYDES

A-8.0 General

The material is mixed with water and the aqueous layer is tested with silver ammonium nitrate solution to determine the presence of aldehydes.

A-8.l Reagents

A-8.l.1 *Silver Nitrate Solution* — Dissolve 1 g of silver nitrate in 20 ml of water.

A-8.1.2 *Ammonium Hydroxide Solution* — 10 percent (*v/v*).

A-8.1.3 *Silver Ammonium Nitrate Solution* — Add ammonium hydroxide solution drop by drop to silver nitrate solution until the precipitate first formed is dissolved completely leaving a clear solution. Do not add excess ammonium hydroxide.

A-8.2 Procedure

Shake 50 ml of the sample with 50 ml of distilled water in a separating funnel for 5 min. Allow the layers to separate and draw off the methylene chloride layer and discard. Collect the aqueous layer in a measuring jar. Take 10 ml of the aqueous layer in a test tube, add 5 ml of silver ammonium nitrate solution and shake well. Keep the test tube in dark for 15 min.

A-8.2.1 The material shall be taken to have passed the test, if no brown colour or turbidity is developed.

A-9 DETERMINATION OF IRON

A-9.0 General

The material is evaporated and the residue is treated with ammonium thiocyanate solution.

A-9.1 Apparatus

A-9.1.1 *Nessler Cylinders* — 100 ml capacity.

A-9.2 Reagents

A-9.2.1 *Concentrated Hydrochloric Acid* – (*see* IS 265 : 1993).

A-9.2.2 *Ammonium Persulphate*

A-9.2.3 *Ammonium Thiocyanate Solution* — approximately 10 percent wt/vol.

A-9.2.4 *Concentrated Sulphuric Acid* — (*see* IS 266 : 1993).

A-9.2.5 *Standard Iron Solution* — Dissolve 0.702 g of ferrous ammonium sulphate [$FeSO_4\ (NH_4)_2SO_4.6H_2O$] in 100 ml of distilled water and 10 ml of concentrated sulphuric acid and dilute with water to 1000 ml mark. Transfer 100 ml of this solution to 1000-ml volumetric flask and dilute again to 1000 ml. One millilitre of this solution is equivalent to 0.01 mg of iron (as Fe). Further dilute 100 ml of this solution to 1 000 ml. One ml of this diluted solution is equivalent to 0.001 mg of iron (as Fe).

A-9.3 Procedure

Dissolve the residue from **A-4.2** with 1 ml concentrated hydrochloric acid and 20 ml water by warming and then make up to 100 ml with water. Take 50 ml of this solution into a Nessler cylinder, add 30 mg of ammonium persulphate and 1 ml of ammonium thiocyanate solution. Carry out a control test in another Nessler cylinder using the same reagents in the same total volume using 2.5 ml of standard iron solution in place of the material.

A-9.3.1 The material shall be taken to have passed the test, if the intensity of the colour produced with the material is not greater than that produced in the control test.

NOTE - Other type of spectrophotometric method may be used, provided slope determination for the specified range is established and accuracy level as specified in this standard is achievable. Alternatively, instrumental method of analysis like AAS, ICP may be used, provided calibration of instrument is done in accordance with detection levels as necessary with requirements of this standard.

A-10 DETERMINATION OF HEAVY METALS

A-10.0 General

Heavy metals are detected by treating with hydrogen sulphide water (as given from **A-10.1** to **A-10.3**) or by alternate test method given at **A-10.4.**

A-10.1 Apparatus

A-10.1.1 *Nessler Cylinder* — 100 ml capacity.

A-10.2 Reagent

A-10.2.l *Hydrogen Sulphide Water* — Pass hydrogen sulphide gas through water and prepare a saturated solution of hydrogen sulphide water.

A-10.3 Procedure — To another 25 ml sample of aqueous extract from **A-9.3**, taken in a Nessler cylinder, add 25 ml of water and 5 ml of hydrogen sulphide water.

A-10.3.1 The material shall be taken to have passed the test, if the colour formed is not greater than the blank similarly prepared.

A-10.4 Alternate Test Method For Heavy Metals Determination

A-10.4.0 Heave metals may also be determined by instrumental methods such as Inductively Coupled Plasma (ICP). Heavy metals shall be reported as lead.

Instrument must be calibrated with certified reference material.

A-10.4.1 Procedure for testing of heavy metals on ICP-OES is as follows:

A-10.4.1.1 *Reagents and Glassware*

a) Ultra pure water; resistivity 18.2 meg.ohm
b) Supra-pure nitric acid.
c) 50 ml volumetric flask- Class A.
d) Micropipette- 100 -1 000 µl.
e) Micropipette- 10 -50 µl.
f) Micro tips.
g) Porcelain basin, capacity 150 ml
h) Water bath.
j) Oven.
k) Lead- Certified reference material, (100 mg/l).

A-10.4.1.2 *Sample preparation*

Take about 100 ml of Methylene chloride sample in to a clean and dry porcelain basin. Evaporate the material over a water bath in a fume hood to practically dryness. Further heat in oven at 105° to 110° C for 30 min. Cool it and add 40 ml ultrapure water. Add 3 ml of concentrated Nitric acid and make up to 100 ml with ultrapure water and mix well.

A-10.4.1.3 *Instrument operating conditions*

Plasma gas flow (l/min)	:	15.00
Auxiliary gas flow (l/min)	:	0.4
Nebulizer gas flow (l/min)	:	0.6
RF power (w)	:	1450
Plasma view	:	Axial
Peristaltic pump flow rate (ml/min)	:	1.5
Calibration	:	Linear
Spray chamber	:	Cyclonic
Nebulizer	:	Meinhard Concentric
Injector ID (mm)	:	2.0 (Alumina)
Quartz torch	:	1-slot

NOTE — For other instrument, follow the procedure as prescribed in respective instrument operation and instruction manual.

A-10.4.1.4 *Procedure*

Open workspace- load method for Methylene chloride details as below:

a) Then, create path for results data.
b) Check and start pump flow, then switch on the plasma.
c) After 10 min of plasma stabilization with wash solvent running, then start the analysis.

Start analysis in the order give below;

1. Blank
2. standards in the order.
3. sample.

Results of the sample will get displayed at the end of analysis in results display window.

A-11 TEST FOR RESISTANCE TO POTASSIUM PERMANGANATE

A-11.0 General

The material is treated with potassium permanganate and examined for its resistance.

A-11.1 Reagent

A-11.1.1 *Potassium Permanganate Solution* — 0.1 percent solution.

A-11.2 Procedure — Take 20 ml of the sample into a stoppered measuring cylinder. To this solution add 0.2 ml of potassium permanganate solution. Mix well and keep aside the cylinder in an ice-bath for 30 min.

A-11.2.1 The material shall be taken to pass the test, if the pink colour does not entirely disappear.

A-12 NESSLER'S REAGENT TEST

A-12.0 General — The aqueous extract of the sample is treated with Nessler's reagent and tested for the presence of ammonia and aldehydes.

A-12.1 Reagent

A-12.1.l *Nessler's Reagent*

A-12.2 Procedure — To another 20 ml of aqueous extract from **A-8.2** taken in a stoppered cylinder, add 5 ml of Nessler's reagent. Shake well and leave for 5 min.

A-12.2.1 The material shall be taken to have passed the test, if no turbidity in yellow, red or black colour is formed.

A-13 TEST FOR RESISTANCE TO SULPHURIC ACID

A-13.0 General

The material is treated with sulphuric acid and tested for the presence of carbonizable impurities.

A-13.1 Reagent

A-13.1.1 *Concentrated Sulphuric Acid — (see IS 266 : 1993).*

A-13.2 Procedure

To 20 m1 of the sample in a stoppered measuring cylinder, carefully add 5 ml of concentrated sulphuric acid. Mix well and keep aside for 5 min.

A-13.2.1 The material shall be taken to have passed the test, if the colour of the acid layer is not more than faint yellow.

A-14 DETERMINATION OF TOTAL IMPURITIES DUE TO METHYL CHLORIDE AND CHLOROFORM

A-14.0 General

The presence and estimation of methyl chloride and chloroform in the sample is determined by chromatographic analysis.

A-14.1 Apparatus

A-14.1.1 *Gas Chromatograph*

Any commercially available chromatograph with a Flame Ionization Detector (FID) may be used. The operating conditions of one of the chromatograph with capillary column are given below:

Column

Material	Fused silica (DB 624)
Length	60 m
Internal diameter	0.25 mm approximately
Film thickness	1.4 micrometer
Stationary phase	cyanopropyl phenyl 94% dimethyl polysiloxane

A-14.1.2 *Microliter Syringe* — 25 microlitre capacity

A-14.1.3 *Recorder* — Chromatogram is displayed on the computer screen.

A-14.2 Reagent

A-14.2.1 *Acetone*

A-14.2.2 *Synthetic Standard of Pure Methylene Chloride,* containing known amounts of methyl chloride and chloroform.

A-14.3 Procedure

A-14.3.1 The operating parameters of the gas chromatograph nitrogen as carrier gas shall be as follows:

	With Nitrogen as a carrier gas
a) Detector block temperature, °C	250 ^{o}C
b) Injection port temperature, °C	230 ^{o}C
c) Column oven temperature, °C	50^{o} C
d) Bridge current	NA
e) Carrier gas pressure, bars	2.1
f) Carrier gas flow rate, m1/min	1.9
g) Sample size, microlitre	1
h) Make up (N_2) flow m1/min	25
j) Hydrogen flow rate, m1/min	30
k) Zero air flow rate, m1/min	300
m) Septum purge Flow rate, m1/min	3
n) Oven Programming	50°C hold 4 min., increase temp. at 5°C/min. upto 150^{o}C

NOTE: The above Gas Chromatographic (GC) conditions are suggestive. However, any GC method having difference in Detector, Column packing material and type (like packed / capillary, diameter, length, film thickness etc), Calibration technique (internal std, external std, area normalization, percent area etc), Carrier Gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

A-14.3.2 *Sample Injection*

Rinse the 25 µl syringe with acetone twice and dry for 2 min or longer. Rinse the syringe twice before filling it with 1 ml of the synthetic standard. Inject the standard solution into the gas chromatograph and obtain a standard chart. Rinse the syringe twice with the material under test and inject 1 µl into the chromatograph. It is imperative that the same injection technique is used and the syringe used in the operation is not used with any other type of solvent. It is very necessary to prevent contamination.

A-14.3.3 *Determination of Response factor (Rf)*

Take neat sample of methyl chloride, methylene chloride, chloroform and prepare standard mixture accurately in neat methylene chloride.

Name of the impurity	Standard Mixture in %
Methyl chloride	0.008 – 0.009
Chloroform	0.008 – 0.009

Inject above standard mixture three times one by one and read the response percentage (%) of Methyl Chloride and Chloroform. Calculate the Response Factor of both impurities in Standard by using given formula,

$$\text{Response Factor of Methyl Chloride or Chloroform} \quad \frac{\text{Actual Concentration (Percent)}}{\text{Area precent from GC}}$$

Calculate the average Response Factor for both Impurities as average of three readings. The Final Response Factor of Methyl Chloride and Chloroform shall be taken for the calculation of impurities concentration in Methylene Chloride.

A-14.3.4 The elution order of the components is as follows:

a) Methyl chloride,
b) Methylene chloride, and
c) Chloroform.

A-14.3.5 *Calculation*

% age of Methyl Chloride = Area % of Methyl Chloride X Response Factor of Methyl Chloride

% age Chloroform = Area % of Chloroform X Response Factor of Chloroform

ANNEX B

(*Clause* 5.1)

SAMPLING OF METHYLENE CHLORIDE (DICHLOROMETHANE), TECHNICAL

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost three-fourths filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the date of sampling and details given under **4.2**.

B-2 SCALE OF SAMPLING

B-2.l Lot

All the containers in a single consignment of the material of the same grade drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

B-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately.

B-2.3 The number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with Table 2.

Table 2 Number of Containers To Be Selected From Lots Of Different Sizes

(*Clause* B-2.3)

Sl No. (1)	Lot Size (2)	Sample Size (3)
i)	*N*	n
ii)	3 to 15	3
iii)	16 to 40	4
iv)	41 to 110	5
v)	111 to 180	6
vi)	181 to 300	7
vii)	301 to 500	8
viii)	501 to 800	9
ix)	801 and above	10

B-2.3.1 These containers shall be selected at random. In order to ensure the randomness of selection, random number tables may be used *(see* IS 4905 : 2015). In case the random number tables are not available, the following procedure may be used:

Arrange all the containers in the lot in a systematic manner and starting from anyone, count them as 1, 2,…. …., up to *r* where *r* is the integral part of *N/n; N* and *n* being the lot size and sample size, respectively. Every r^{th} container thus counted shall be withdrawn to constitute the sample.

B-3 INDIVIDUAL SAMPLES AND COMPOSITE SAMPLE

B-3.1 From each of the containers selected according to **B-2.3**, a representative portion of the material, about 300 ml in volume, shall be drawn. From each of these individual portions, an equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample not less than 1 000 ml in volume. The composite sample shall be transferred to clean bottles and labelled with full identification particulars of the sample.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of the characteristics given in Table 1 shall be carried out on the composite sample.

B-5 CRITERIA OF CONFORMITY

B-5.1 For declaring the conformity of the lot to the requirements of all the characteristics, the test results on the composite sample shall meet the corresponding requirement specified.

ANNEX-C

(*Foreword*)

IS No.	*Title*
265 : 1993	Hydrochloric Acid — Specification (*fourth revision*)
266 : 1993	Sulphuric Acid — Specification (*third revision*)
878 : 2008/ ISO 4788 : 2005	laboratory glassware — graduated measuring cylinders (*second revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
2837 (Part 2) : 1977	Specification of porcelain crucibles and basins : part 2 basins (*first revision*)
4825 : 1982	Specification of liquid-in-glass solid-stem reference thermometers (*first revision*)
4905 : 2015/ISO 24153 : 2009	Random sampling and randomization Procedures (*first revision)*

ANNEX D

(*Foreword*)

COMMITTEE COMPOSITION

Organic, Chemicals Alcohols and Allied Products Sectional Committee PCD 09

Organization	*Representative(s)*
Chemical Engineering and Process Development Division, NCL Pune	Dr C. V. Rode (***Chairman***)
All India Distilleries Association (AIDA), New Delhi	Shri V. N. Raina
BASF India Limited, Mumbai	Shri Kiran Bhat Shri Hemal Berawala (*Alternate*)
CDRI, Lucknow	Dr Sanjeev Kanojiya
Central Revenues Control Laboratory, Delhi	Dr T. A. Sreenivasa Rao
Chemical and Petrochemicals Manufacturers Association (CPMA), New Delhi	Shri Kamal Nanavaty
Deepak Fertilizer, Pune	Dr Satish Chand Saini Shri Suresh Amle (*Alternate*)
Deepak Phenolics Limited, Bharuch	Dr S. Samal Shri Nr Sandipkumar Pancha (*Alternate*)
Dow-Corning India Limited, Mumbai	Shri Abraham Barrett Shri Ritesh Gulabani (*Alternate*)
Indian Chemical Council, Mumbai	Dr. Mritunjay Chaubey Shri J. I. Sevak (*Alternate*)
Jubilant Life Sciences Ltd, Noida	Shri Hari Mohan Lohani
National Chemical Laboratory, Pune	Dr Udaya Kiran Marelli
UPL Limited, Mumbai	Mr M. D. Vachhani, Gm (Qa)
Alkyl Amines Chemicals Ltd, Mumbai	Shri S. V. Nikumbhe Shri Sameer Katdare (*Alternate*)
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	Shri K. L. Raphael Shri Kirti Gajjar (*Alternate*)
Godavari Biorefineries Ltd, Mumbai	Shri Shanul Laxmanrao Pagar Ms Wani A. J (*Alternate*)
Gujarat Narmada Valley Fertilizers Company Limited, Gujrat	Dr M. J. Kapadia Shri P. R. Desai (*Alternate*)
Hindustan Organic Chemicals Ltd (HOCL), Mumbai	Shri Deleep Kumar K. Shri V. Mohan (*Alternate*)
India Glycols Limited, Uttarakhand	Shri S. R Soni Shri Alok Singhal (*Alternate*)
Laxmi Organic Indusrties, Mumbai	Shri J. P. Suryavanshi Dr Vijay S. Mishra (*Alternate*)
Ministry of Chemicals and Fertilizers, New Delhi	Shri O. P. Sharma Shri Varun Singh Poonia (*Alternate*)

Organization	*Representative(s)*
National Test House, Ghaziabad	SHRI DEBASHIS SAHA DR GOPAL KRISHAN (*Alternate*)
Reliance India Limited, Mumbai	SHRI K. K. SREERAMACHANDRAN SHRI VASANT WARKE (*Alternate*)
BIS Director General, New Delhi	SHRI N. K. BANSAL, SCIENTIST 'F' AND HEAD (PCD), [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary

SHRI CHANDRAKESH SINGH
SCIENTIST 'C' (PCD) BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: PCD 09 (14902).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402
Website: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	2323 7617 2323 3841
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi KOLKATA 700054	2337 8499, 2337 8561 2337 8626, 2337 9120
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg CHANDIGARH 160019	265 0206 265 0290
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	2254 1216, 2254 1442 2254 2519, 2254 2315
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093	2832 9295, 2832 7858 2832 7891, 2832 7892

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. COIMBATORE. DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. JAMMU. JAMSHEDPUR. KOCHI. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi